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Reuse of residual sludge from stone processing: differences and similarities between sludge coming from carbonate and silicate stones—Italian experiences

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Reuse of residual sludge from stone-processing: differences and similarities between sludge coming from carbonate and silicate stones. Italian experiences

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Abstract

Residual sludge coming from dimension stone working activities represents a serious environmental and economic problem for both Stone Industry and community. Indeed, most of time, residual sludge is landfilled because of the difficulties to recover it; such difficulties are mainly connected to local legislation and to a lack of proper protocols.

In general, two different categories of sludge can be identified: residual sludge coming from carbonate rocks (CS) and those coming from silicate rocks (SS). Both of them are characterised by a very fine size distribution. CS is mainly made up of the same compounds of the processed stones (i.e. marble, limestone, travertine). On the contrary, SS is characterised by high heavy metal content, due to the composition of the tools employed during processing activities and to the original rock characteristics. Furthermore, Total Petroleum Hydrocarbon content can often be recognized in residual sludge.

In general, residual sludge, management of which in Italy is administered in accordance with ILD 152/06, can be used as waste for environmental restoration or for cement plants. Several researches investigate possible reuses of residual sludge, after a proper processing phase, as new products. Such “new products” should be certified not only on the basis of their technical and physical characteristics but also by means of appropriate chemical analyses to guarantee that the products are not polluted.

The aim of this research is to evidence that, on the basis of a correct sludge **characterization, treatment and management**, it is possible to produce **Secondary Raw Materials** (filler, etc...) or **New Products** (artificial soil, etc...), in order not to waste “**sludge resources**”. Some examples from Italian experimentations are here reported, focusing on the treatment and recovery of SS and CS.

Glossary

SRM = Secondary Raw Materials

NP = New Products

CS = sludge from carbonate rocks

SS = sludge from silicate rocks

GSS = sludge from gangsaw using abrasive steel shot

DBC = sludge from multi diamond-saw block cutter

MS = mixed sludge

TPH = Total Petroleum Hydrocarbon

U = Uniformity

ILD = Italian Legislative Decree

IL = Italian Law

a.s. = as such

NA = Not available

1 Introduction

Stone industry is one of the most important productive sectors on the global market, and Italy is still one of the international leaders as for dimension stone production and transformation. The overall turnover of the production chain (quarries, working plants, laboratories, shops, etc...) was nearly 40 billion € in 2011, almost 2 % of the Italian Gross Domestic Product – GDP. In addition, Italian dimension stone exploitation during 2012 shows an export increment of 9.8 % from 2011 and an import decrement equal to -6 % (from 2011 to 2012) (Dino et al. 2014). At present, Italian quarry industry is characterized by incertitude due to global crisis in the stone sector.

Quarries and working plants produce huge amounts of waste, often landfilled, that show a great potential for recovery (Luodes et al. 2012). Fine fraction (residual sludge), coming from working activities (mainly stone cutting and sawing), is the most difficult fraction to recover. Residual sludge (EWC code 01 04 13) is represented by a very fine material (see title 2.2). that can be used, as “waste” in accordance with the ILD 152/06, either as filler for environmental restoration or as feeding material mainly for cement production (ILD, 2006). Costs connected to residual sludge management as waste to dump may represent more than 3% of the operating costs of dimensional stone working plants, with a consequent significant repercussion on company economic balances. In addition to management costs, residual sludge disposal activities contrast the EU principles of waste recycling (Dino et al. 2013).

Thus, residual sludge management still represents a problem for companies and society at large, due to its high management costs and to the necessity of a proper recovery, other than landfill (“Waste recycling” together with “Resource preservation” are two important pillars in EU politics and big efforts are required in finding new solutions, other than waste burial, for residual sludge management).

The present paper develops issues as:

- comparison between the characteristics of the two main sludge categories (silicate sludge – SS, and carbonate sludge - CS)
- environmental problems connected to sludge management
- potential applications for SS and CS, highlighting the most promising ones (to appreciate which recovering activities are more promising, a general overview about the researches developed in the last decades is indispensable: see title 1.1).
- problems connected to “new products” certification and selling
- some suggestions for executive protocols to boost their systematic recovery.

Sate of the art

1.1 Silicate sludge

Several researches about SS were performed during the 70s and the 80s; these researches were targeted mainly to economic issues and to the improvement of the technical performances of the sawing activities [VV.AA., 1987 and 1988], and in particular:

- energy consumption linked to sawing and working activities;
- problems connected to recovery and recirculation of the abrasive pulp (optimization of grit);
- productivity improvement: increase of the feed rate, yield of both the saw blades and abrasive slurry.

During the 90s (the golden age as for the so called “commercial granites”), the studies about residual sludge were essentially addressed towards three different sectors:

- employment as waterproofing material for municipal landfills (Bertolini and Celsi, 1989; Frisa Morandini and Verga, 1990). This solution, if technically possible, can cause some problems connected to the possible leaching of heavy metals from sludge (and it contrasted the regulations in force). Some recent researches (Dino et al, 2013) demonstrate that, according to Italian Legislation (ILD 36/2003), sludge as such (a.s.) and SS mixed with bentonite clay (5-10%) show the requested hydraulic permeability features for dump covering and dump waterproofing. However, the environmental compatibility is the necessary condition to think about such a recovery;
- feldspar and quartz recovery to use in ceramic and glass industries (Curreli et al., 1992; Sassone & Danasino, 1995). The results of those researches was technically promising but too expensive;
- making sludge inert applying the same technologies employed in ceramics and glass industries (Pelino et al., 1998). Such researches have been recently taken up, showing promising results, from Rincón and Romero [2010].

At the end of the 90s, and in the following years, other potential recoveries were investigated. In particular, several researches about the reuse of sludge for land reclamation were carried out. Such researches are based on the premises that quarrying activities require an obligatory phase of environmental rehabilitation of both dumps and quarrying areas, and sludge, properly treated, could be used as “new soil” for these purposes. Several studies investigate the use of different materials, other than topsoil, to employ for quarry rehabilitation (Burrigato et al. 1999; Barrientos et al. 2010; Sivrikaya et al. (2014). In general, a direct agricultural application of sludge is seemingly hindered by their low chemical and physical fertility but they can be treated and recovered to obtain a cultivation substrate.

Sludge treatments to produce “soil” and “filler” for environmental application have been carried on in the last 15 years, employing sludge coming from working activities on silicate rocks (SS). The very fine size distribution of residual sludge, in association with the low organic matter content and high pH, make these materials unusable a.s. in an agricultural-rehabilitation context. Thus, these materials have been mixed to other compounds:

- to compost, shredded pruning material (green manure), and top soil, to obtain “artificial soil” from bioremediation process (Dino et al. 2006, 2014);
- to coarse materials to produce filler for environmental application (Dino et al 2013, 2015);
- to sand, compost and peat to obtain “artificial soil” (Dino et al 2013, 2015).

The results of these experimentations were promising both at a laboratory and at “in situ” scale (i.e. see the environmental rehabilitation of a quarry in Luserna Stone quarry basin: Cava del Tiglio-Pra del Torno, Rorà – TO, NW Italy), reported in Dino et al. 2014 or the results connected to the use of mixed sludge as filler material for land reclamation (Dino et al 2015).

1.2 Carbonate sludge

The disposal of microfine marble sawdust contained in marble slurry waste currently represents an additional economic burden for dimension stones companies (Careddu et al, 2014). This is because the sawdust is considered less important in comparison with the crushed calcareous aggregate produced in stone processing plants. Although the chemical compounds of the limestone microfine dust, which have been determined with laboratory tests and trials, have ruled out the presence of polluting materials (Careddu et al. 2009) demonstrating that these materials are affected by a virtually nil level of pollution, dewatered sludge are considered a problem. For this reason, both businessmen and politicians should see carbonate microfine material from another point of view. A concrete example can be the one of Orosei (Sardinia, Italy) where a polishable limestone (commercially known as “Orosei Marble”) is quarried and processed. In that producing area, local authorities and industrial associations are now reasoning in order to find an adequate solution, which could respect the stricter environmental laws (ILD, 2006). The producers together with the Municipality of Orosei, which founded a Consortium aimed to manage of waste disposal to the landfill, are planning to transform such landfill into a Centre for stone materials aimed at secondary processing, training sessions on different subjects related with Dimension Stone sector and industrial tourism by mean of a historical route (Careddu et al., 2013).

However, state of the art in recovery and utilisation of calcareous sawdust is mainly aimed in uses as substitute for more expensive ingredient in the building sector. Indeed, bibliography about the re-use of fine dust resulting from the processing of marble mainly pertains to products such as: concrete (Almeida et al., 2007; Felekoglu, 2007; Topçu et al., 2009; Marras et al., 2010b; Gencel et al., 2012; André et al., 2014), ceramic (Díaz and Torrecillas, 2007; Saboya et al., 2007; Montero et al., 2009a,b; Marras et al., 2010a; Devant et al., 2011), other building products (Lee et al., 2008; Galetakis et al., 2012). Other limestone sawdust uses regard the acid mine drainage (Barros et al., 2009). Sort and Alcañiz (1996) studied the effects of sewage sludge additions to control erosion in limestone quarries. They reported a positive effect on soil physical properties in general and on soil loss in the plots treated with the sludge.

2. Sludge characterization

2.1 Origin

As introduced in title 1, it is possible to individuate two different categories of sludge: CS and SS. Moreover, SS can be split in three different sub-categories, depending on the way they are produced: sludge from gang saw using abrasive steel shot (GSS), sludge from multi diamond-saw block cutter (DBC), and mixed sludge (MS) - from both gang saw and block cutter.

These materials derive from the stone-processing activities, which are quickly described as follow (Fig. 1). Granite “standard” blocks are sawn in slabs by mean of traditional steel-shot gang saw or modern multi-wire machines. Slabs are then sold a.s. or subjected to a finishing treatment (especially as gauging-smoothing-polishing, flaming). Both raw and finished slabs are then cut in tiles by mean of machines equipped with diamond disk (jib saws, bridge saws, continuous multi-disk milling machines). Granite “non-standard” shaped blocks are sawn by block-cutters into strips, which are then cut in size (tiles) and, eventually subjected to a finishing treatment.

The production cycle for marble blocks is similar to the previous one; the strategic difference is that the marble production process is fully based on the use of diamond technology. “Standard” block sawing is carried out by multi-blade gang saws equipped with diamond blades. If necessary, marble blocks are previously subject to squaring (by single-diamond-wire machine) when they arrive from the quarry not squared enough to guarantee good gang saw fill (Primavori, 2008). Figure 1 summarizes the production cycles of the companies monitored during this study.

INSERT FIG.1

The turbid waters produced during cutting and polishing phases have to be treated. It is fundamental to separate the solid fraction from water because, on the one hand, water must not be wasted (treated water is collected and reused during cutting and polishing phases), and on the other hand, dewatered sludge is lighter to manage, and costs connected to transport and landfilling activities depend on the weight of the material. The treatment on turbid water is represented mainly by three different alternative activities:

- thickening using big bags (fig. 2.a)
- thickenings by means of settling basins/tanks (fig. 2.b)
- filter pressing activity for SS and CS (fig. 2.c,d)

INSERT FIG.2

2.2 Physical characterisation

The physical characteristics of residual sludge depend on the original material and on the working activities. The present research shows results connected both to CS and SS, and, in particular, CS (general) and SS (GSS, DBC and MS).

As for the CS (Fig. 3.c), the authors carried out sampling of sludge in four factories, all of which followed different production cycles; sludge samples were collected from the filter press outlet port (where dewatered sludge were stockpiled). Grain size analysis was conducted on the dry solid cake produced by the filter press of all processing plants using a Sedigraph 5100 Analyser.

As for SS, six different materials were sampled: two from DBC (Fig. 3.b), two from GSS (Fig. 3.a), and two from MS. The six samples came from three different working plants, in which granites and gneisses from Verbano Cusio Ossola quarry basin (VCO - NW Piedmont) were cut and polished. The grain size distribution was measured with sieve analysis according to ASTM standards (ASTM D421-85(1998); D422-63(1998); D1140-00; D2217-85(1998)).

INSERT FIG. 3

Both CS and SS are characterised by a very fine size distribution (silt-clay dimensions; Fig. 4 and Tab. 1): 40 % of the solid fraction is inferior than 25 μm ; sludge is an incoherent material characterized by asphyxial properties.

INSERT FIG. 4 AND TAB. 1

Looking at Fig. 4 and Tab. 1, some considerations can be underlined:

- 1) CS coming from working activities employing diamond tools analyzed in this research (coming from Orosei limestone) show a high uniformity and a minor d_{50} ;
- 2) CS coming from honing and polishing line show a size distribution little bit different from the CS coming from working activities employing diamond tools: higher d_{50} and U;
- 3) SS (GSS, DBC and MS deriving from granites and gneisses from VCO quarry basin) show a size distribution generally coarser than the one of CS (higher d_{50} and U).

As for SS, it has to be highlighted that these materials show a very low permeability: the value connected to hydraulic conductivity k is very low. Hydraulic conductivity was evaluated using falling head permeability tests (ASTM D 5084) with modified triaxial cell (two samples of the original six). Falling head permeability tests highlight a k equal to $2.3 \cdot 10^{-8}$ m/s for DBC, $2.9 \cdot 10^{-8}$ m/s for GSS and $9.2 \cdot 10^{-9}$ m/s for MS. (Dino et al 2013).

2.3 chemical characterization

SS is characterised by high heavy metal and Total Petroleum Hydrocarbon (TPH) content, in particular:

- Heavy metals derive from abrasive gangue (Fe, Ni, Cr) associated to lime (as antioxidant) present in steel shot gang saw, and from metal powders used in diamond alloys for diamond cutting tools (Co, Fe), present in diamond frame shot.

The three sub-categories characterizing SS (GSS, DBC, MS) show different problems connected to heavy metal content, indeed on the one hand GSS is characterised by a high percentage of Ni, Cr, Cu, etc., on the other hand DBC is characterised by Co and Cu high content (Tab. 2).

Unlike granite, marble processing allows a significantly higher yield of diamond tools (Careddu and Marras, 2015). The reason is strictly linked to the lack of silicate minerals (such as quartz and feldspars, highly abrasive) in carbonate rocks. Therefore, the carbonate sludge is affected by a virtually nil level of heavy metals (Tab. 2), deriving from the wear of diamond-tools, when compared with similar sludge produced by sawing/cutting/polishing granite.

All waste parameters were measured in the eluates; the results of the leaching tests were then compared with the threshold values. Their concentrations were below the limit specified in the table in Italian Legislative Decree no. 186 (2006)

- TPH content is connected to mineral oils, lubricants (C12-C40), etc., coming from oil machines losses. These substances are immiscible with water, and require a specific degradation process (i.e. Bioremediation process) if they are not to pollute the material (Dino et al. 2014).

Heavy metals (Fe, Cr, Ni, etc...) can be separated and recovered using magnetic or gravimetric separation. Indeed, some experiments were carried out to test the possibility to recover magnetic/metallic fraction from GSS and use this fraction, together with "clean sludge", for SRM exploitation. In general, it has to be underlined that the Fe is mainly concentrated in the > 100 mesh fractions. It could therefore be possible to

perform a “size cut” for the >100 mesh fractions, and a consequent wet magnetic separation. (Dino et al. 2003);

In general, CS is composed mainly by the same compounds of the processed stones (marble, limestone, travertine); it becomes very interesting, from an economic point of view, when it has a CaCO_3 grade > 95 %.

INSERT TAB. 2

3 Discussion

To guarantee the systematic and convenient recovery of **sludge materials** as SRM, both quarry and stone-working plants should be planned for the purpose of:

- wastewater should be collected in order to be diverted to a separate section of the water-treatment plant;
- the disposal area for marble and silicate scraps should be projected as a centre for stone materials aimed at secondary processing (as already cited in 1.2). Scraps produced from quarrying and stone processing should be orderly located in a storage outdoor area, in view of their secondary processing.

Furthermore, to produce NP both from SS and CS, it is necessary to think about several actions, such as:

- a proper treatment depending on the kind of reuse. A treatment-activities protocol should be forecasted in order to produce each NP. Moreover, such protocol has to be shared with Public Authorities, which has to authorise the activities connected to treatment plant.
- different production lines for GSS, DBC, MS, in order to guarantee feeding materials to treatment plant, characterized by similar size distribution, heavy metal content, etc.... These different lines are fundamental to separate waste with different characteristics, applying different treatment activities, try to maximise the recovery of each sludge category (i.e. as for GSS a magnetic or hydrogravimetric separation is fundamental to separate heavy metals connected to abrasive steel shot).

Also for CS, it is strategic to stress that different processes can produce sludge with different properties: i.e. differences in colour between the sludge deriving from block sawing or/and slab cutting and those produced by slab polishing (Careddu et al., 2014). Therefore, in order to match better the standard requirements for CaCO_3 by improving brightness, colorimetric and chemical properties of sawdust, some changes in the sewage line may be required.

- the environmental protection has to be guaranteed; thus, adequate physical and chemical analysis (pH, size distribution, hydraulic conductivity, etc.) and monitoring activities must be forecasted on the raw materials feeding the treatment plants which produce NP, and on the NP themselves.
- a market ready to accept NP obtained from Waste treatment. It is necessary to inform and sensitise the civil society about the necessity to accept and use product coming from waste treatment (*End of Waste* Criteria; http://ec.europa.eu/environment/waste/framework/end_of_waste.htm).
- a joint venture between public authorities and private companies to work on a shared legislation and on the adoption of proper technical documents for materials to employ for Public Works and infrastructures; the inclusion in such documents of products coming from waste recovery is fundamental to boost the use of NP from sludge recovery (filler materials, artificial soil and waterproof materials).

Currently limestone dust, recovered from marble sawing and processing slurry, has not found very economically feasible industrial usage; researches have been mainly focussed on the re-use of marble micro-fine sawdust in low cost applications (as building materials are). A more sustainable alternative is the re-use of the microfine dust as a by-product; in this way, companies can cover landfill costs with revenues generated with the sale of these goods. Taking into account that the global economic crises has affected mainly construction and building sectors, it appears more strategic to focus on the uses of CaCO_3 in high value-added products: a focused research on the recycle of marble dust for higher-end products such as paper, rubber, pharmaceuticals has to be encouraged.

It should be noted that the market value of CaCO_3 depends essentially on three properties: purity, particle size and brightness (Prescott and Pruett, 1996); currently dry GCC with 0.5 mm mean particle size has a Free on Board price of about 100 - 112 €/t (Marras, 2011). Previous studies (Careddu et al, 2014; Careddu and Marras, 2015) based on Orosei Marble producing area (Italy) proved the features of microfine calcareous dust can meet the requirements of the industrial top-of-the-range products. A recent research (still not published) demonstrated that microfine calcareous dust can be successfully used in tyre mixtures. Further future developments in the use of CaCO_3 -sludge produced during stone-working should be focused on paper production, cosmetics and animal feed sectors. Particularly, the last two sectors have CaCO_3 requirements very strict; so, it is not easy to match them. However, researcher should try to meet those requirements –using mineral processing techniques- in order to give the maximum added-value to CaCO_3 . In

order to match these last statements, is strategic to note that, following the environmental law ILD 161 (2012), the introduction of biodegradable flocculants in waste-water treatment should be encouraged; indeed, the presence of traditional anionic flocculants, which have acrylamide or polyacrylamide, is considered detrimental for the reuse of the stone waste. However, the last Italian Law, about Green Economy (IL 2016), has overcome this problem.

Another separation method to be studied is the technology based on cyclones, which can guarantee a good separation of sludge into different grain size classes.

4 Conclusion

As described in the paper, several researches during the last decades investigated the possible reuse of residual sludge but, at present, there is no evidence of its systematic recovery as Secondary Raw Materials (SRM) or for NP production (recycled product). On the basis of the results arising from these researches, it is possible to highlight its recovery, after a proper treatment, mainly as: landfill waterproofing material, filler material for civil works, artificial soil for land rehabilitation and high value added products (mainly from carbonate rocks). SRM and NP have to be certified not only on the basis of their technical and physical characteristics but also by means of appropriate chemical analyses to guarantee that NP are not polluted, avoiding potential environmental pollution for water, soil and air. It is important to establish operative protocols:

- to forecast periodical test on the raw materials and on the NP
- to guarantee the possibility of using “alternative products” for Public and Private works (infrastructures, soil reclaim, etc., mainly from SS) When environmentally guaranteed, NP employment, such as “artificial soil to use for quarry rehabilitation, respect the “cradle-to-cradle principle”: all the exploited material would be sold as products and the waste could be employed for the environmental rehabilitation of the “cradle quarry site”. Such kind of application guarantees the re-use not only of “quarry” and working plants waste, but also of compost, peat and organic fraction in general.
- to produce high value products (mainly from CS) in order to boost the use of residual sludge in new productive cycles.

The use of SRM and NP (from recycling activities) is also one of the main issues of the recent EU directive about Circular Economy, which aims at guaranteeing the use of waste in different productive cycles in order to reach the zero-waste production.

In general, the production of sludge, which characterized the 80s up to the beginning of the XXI cent., caused huge problems connected to its management and landfilling. From the beginning of the years 2000s the production of sludge has been decrementing. This reduction is mainly connected to the global crisis, which characterized the stone sector, and in particular the one connected to granites (commercial categories, UNI EN 12440) exploitation. Due to the crisis, stone materials have been underexploited with a consequent reduction in sludge amount production. Furthermore, granites are less interesting for architects, and they are not employed in building industry as before. However, a slight upswing in stone sector has to be noticed (Montani, 2014). Moreover, the problems caused by heavy metal content is less incisive; the decrement of heavy metal content is close linked to the wider employment of multi-wire machines instead of traditional gang saw using abrasive steel shot (Careddu and Cai, 2014).

Even if problems connected to sludge management seem to be decremented in the last decade, due to crisis in stone sector and to development in processing techniques, such waste are still landfilled. As introduced, “waste recycling” is EU mandatory, thus, sludge recovery in new products production has to be the target of companies, researchers and Public Authorities. The reuse of waste to obtain SRM and NP is one of the goals of Environment and Raw Materials Societal Challenges EU programs (H2020 calls): thanks to the systematic recovery of such a kind of waste the EU fundamental principles of “waste recovery” and “resources preservation” are carried out.

Future researches shall be developed trying to bridge the gap about residual sludge characteristics and about the best techniques and methodologies to apply for residual sludge recovery. For instance, more info about bulk density, withdrawal limit and hydraulic conductivity are surely to be known better. Moreover, protocols about the best methodologies to test sludge characteristics should be developed. At last but not least, also protocols connected to sludge treatments (i.e. raw materials, dressing activities, periodical tests) have to be carried out.

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Table 1: summing up of the size distribution of residual sludge, depending on sludge characteristics (rocks and working tools)

		d_{50} [μm]	U
Residual sludge from carbonatic rocks (CS)	Coming from working activities employing diamond tools (average)	3,8	4,8
	From honing and polishing line	5,2	6,3
Residual sludge from silicatic rocks (SS)	From gang saw using abrasive steel shot (GSS) (average)	7,7	10,6
	From multi diamond-saw block cutter (DBC) (average)	8,3	10,0
	Mixed sludge (MS) (average)	8,2	10,3

Table 2: residual sludge main chemical characteristics (Blue: not negligible concentrations, below the limits of the law; Yellow: concentrations over the limits for residential areas; Red: concentrations over the limits for industrial areas) (according to D.Lgs. 152/06, Italian Legislation)

Samples	As (mg/kg)	Co (mg/kg)	Cr tot (mg/kg)	Cr VI (mg/kg)	Hg (mg/kg)	Ni (mg/kg)	Cu (mg/kg)	Zn (mg/kg)	TPH (mg/kg)
GSS1	12.0	10.7	65.3	26.3	< 0.5	35.6	114.9	64.8	43.5
GSS2	14.2	10.0	69.6	28.1	< 0.5	37.1	131.6	65.0	73.8
DBC1	5.7	80.3	5.5	1.2	0.7	15	46.7	59.8	49.6
DBC2	4.1	91.0	< 5.0	1.4	0.8	2.7	33.3	51.1	40.3
MS1	10.1	12.2	59.1	50.6	0.6	40.4	86.6	61.6	77.9
MS2	9.3	10.1	50.6	36.9	< 0.5	47.3	91.3	57.9	41.4
CS1	< 5	< 1	< 20	NA	NA	< 20	< 10	< 30	NA
CS2	< 5	< 1	< 20	NA	NA	< 20	< 10	< 30	NA

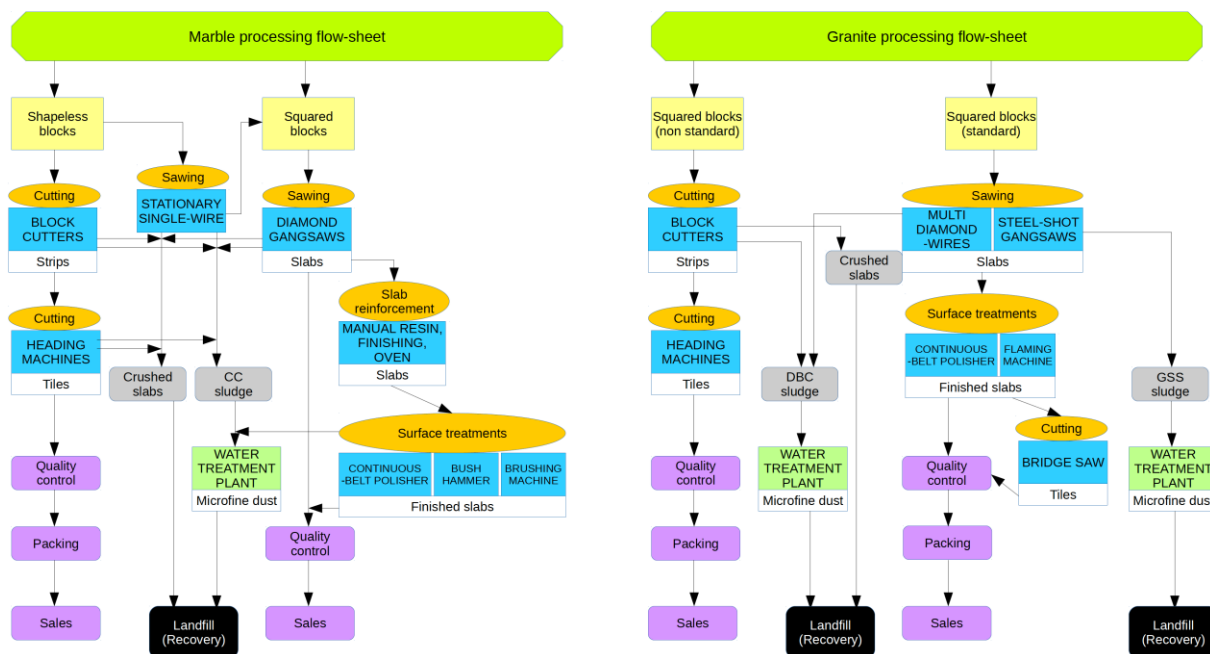


Figure 1: marble and granite processing flow-sheets

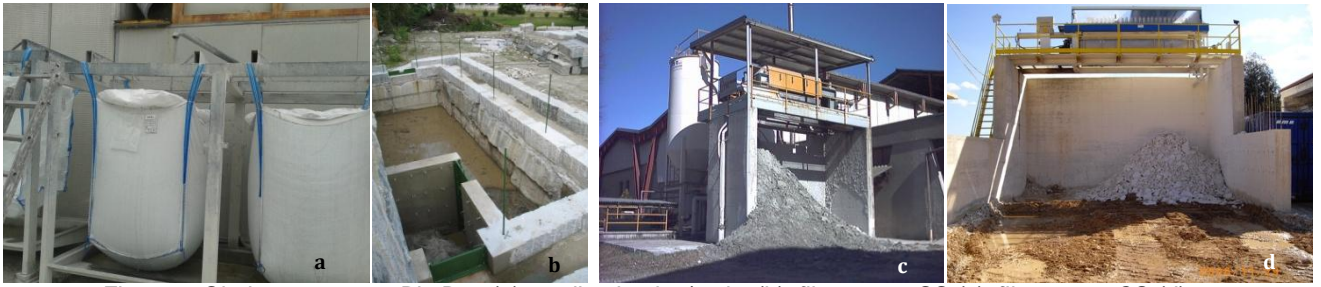


Figure 2: Sludge treatment: Big Bag (a); settling basins/tanks (b); filter-press SS (c); filter-press CS (d)



Figure 3: GSS dry samples (a); DBC dry samples (b); CS dry sample (c)

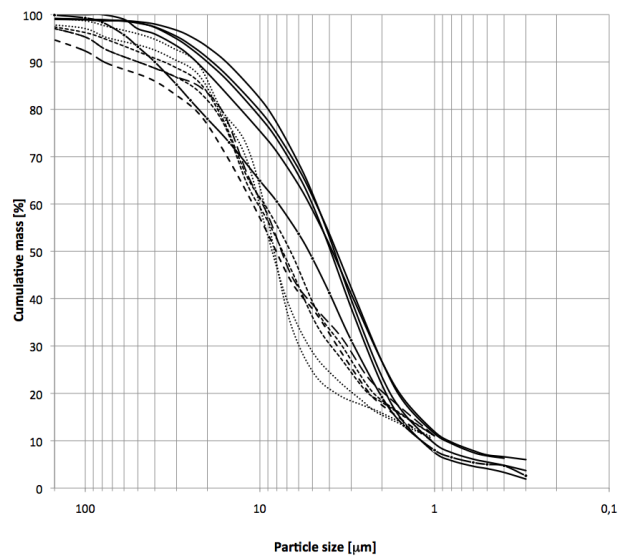


Figure 4: residual sludge particle size distribution. Continuous lines: CS from sawing/cutting process; continuous line with black dots: CS from honing and polishing line; long-dashed lines: GSS; short-dashed lines: MS ; dotted lines: DBC.